IS 6798 (1997): Octyl Gallate, Food Grade [FAD 8: Food Additives]

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भारतीय मानक

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(पहला पुनरीक्षण)

Indian Standard

OCTYL GALLATE, FOOD GRADE — SPECIFICATION

(First Revision)

ICS 67.220.20; 71.080.70

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

October 1997

Price Group 2

AMENDMENT NO. 1 FEBRUARY 2006
TO
IS 6798 : 1997 OCTYL GALLATE, FOOD GRADE — SPECIFICATION

(First Revision)

(*Page* 1, *Table* 1) — Delete Sl No. (viii) and renumber the subsequent serial numbers.

[*Page* 2, *Table* 1, *Sl No.* (ix), *col* 3] — Substitute '10' *for* '30'.

(FAD 8)

Reprography Unit, BIS, New Delhi, India

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Food Additives Sectional Committee had been approved by the Food and Agriculture Division Council.

With the increased production of processed foods, manufacturers have started adding a large number of substances, generally in small quantities, to improve the appearance, flavour, texture or storage properties, etc, of the processed foods. As certain impurities in these substances have been found to be harmful, it is necessary to have a strict quality control of these food additives. A series of standards was, therefore, prepared to cover purity and identification of these substances. These standards would help in checking purity, which requires to be checked at the stage of manufacture, for it is extremely difficult to detect the impurity once these substances have been added to the processed foods. Besides, these standards are intended to guide the indigenous manufacturers in making their product conform to specifications that are accepted by scientists, health authorities and international bodies.

Use of octyl gallate, food grade is permitted under the *Prevention of Food Adulteration Rules*, 1955 as an anti-oxidant in edible oils and fats except *GHEE* and butter. Its chemical names are octyl gallate and n-octyl ester of 3, 4, 5-trihydroxybenzoic acid. Its empirical formula is $C_{15}H_{22}O_5$. Its molecular mass is 282.34. Structural formula of octyl gallate is:

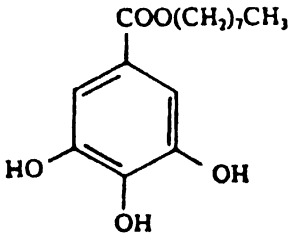

FIG. 1 STRUCTURAL FORMULA

This standard was first published in 1972. The Standard is being revised to make the following additions/changes:

a) To provide a separate clause for description including the solubility property to keep it in line with Food Chemical Codex NRC.

b) To upgrade the standard by providing limits for heavy metals, chlorinated organic compounds, free acid and absorption.

c) To provide for marking instructions for storage and expiry/best before date.

d) To update referred standards.

In the preparation of this standard, considerable assistance has been derived from Compendium of Food Additive Specifications, Volume 2, Joint FAO/WHO Expert Committee on Food Additives (JECFA), 1992 and this standard is harmonized with the standard of FAO/WHO.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values' (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

OCTYL GALLATE, FOOD GRADE — SPECIFICATION

(First Revision)

1 SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for octyl gallate, food grade.

2 REFERENCES

The following Indian Standards contain provisions which through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
1699 : 1995	Methods of sampling and test for synthetic food colours (*second revision*)
2362 : 1993	Method for determination of water by the Karl Fischer Method (*second revision*)

3 DESCRIPTION

Octyl gallate shall be white to creamy-white odourless solid which may have a slightly bitter taste. The material shall be insoluble in water and freely soluble in ethanol, ether, propylene glycol and fat.

NOTE — The solubility is intended only as information regarding approximate solubility and is not to be considered as a quality requirement and is of minor significance as a means of identification or determination of purity, and dependence must be placed on other specifications.

4 REQUIREMENTS

4.1 Identification Tests

4.1.1 Melting range shall be 99 to 102°C after drying at 90°C for 6 hours.

4.1.2 Add 1 ml of ammonium hydroxide to 5 ml of one percent ethanolic solution of octyl gallate. A pink to red colour shall appear.

4.2 The material shall also conform to the requirements given in Table 1.

Table 1 Requirements for Octyl Gallate, Food Grade

Sl No.	Characteristic	Limit	Method of Test, Ref to	
			Annex of This Standard	IS 1699
(1)	(2)	(3)	(4)	(5)
i)	Purity as $C_{15}H_{22}O_5$ percent by mass, *Min*	98.5	A-1	—
ii)	Moisture, percent by mass, *Max*	0.5	A-2	—
iii)	Sulphated ash, percent by mass, *Max*	0.05	A-3	—
iv)	Chlorinated organic compounds, (as chlorine), mg/kg *Max*	100	A-4	—
v)	Free acid (as gallic acid), percent by mass, *Max*	0.5	A-5	—
vi)	Specific absorption at 275 nm *Min* 1%	375	A-6	—
	Max 1 cm	390		
vii)	Arsenic (as As), mg/kg, *Max*	3	—	15
viii)	Lead (as Pb), mg/kg, *Max*	10	—	15
ix)	Heavy metals (as Pb) mg/kg, *Max*	30	—	16[1)]

[1)] The quantum of sample to be taken for test shall be 1 g.

5 PACKING, STORAGE AND MARKING

5.1 Packing

The material shall be securely packed in well-filled containers with minimum access to light and air. The containers shall be such as to preclude contamination of the contents with metals or other impurities.

5.2 Storage

The material shall be stored in a cool and dry place so as to avoid exposure to heat.

5.3 Marking

Each container shall be marked legibly and indelibly marked with the following information:

a) Name of the material including the words 'Food Grade';
b) Name and address of the manufacturer;
c) Net content when packed;
d) Batch or code number;
e) Instructions for storage;
f) Expiry/Best before date; and
g) Any other requirements as specified under the *Standards of Weights and Measures (Packaged Commodities) Rules*, 1977 and *Prevention of Food Adulteration Rules*, 1955.

5.3.1 *BIS Certification Marking*

The containers may also be marked with the BIS Standard Mark.

5.3.1.1 The use of the Standard mark is governed by the provisions of *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

6.1 Representative samples of the material shall be drawn according to the method prescribed in 4 of IS 1699.

7 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

(*Table* 1)

ANALYSIS OF OCTYL GALLATE

A-1 PURITY

A-1.0 Two methods, that is, spectrophotometric and bismuth nitrate, have been specified. Either could be used depending upon the facilities available.

A-1.1 Spectrophotometric Method

A-1.1.1 Dry a suitable quantity of sample in an oven at 90°C for 6 hours. Cool it in a desiccator. Prepare a solution of a dried sample in 80 percent ethanol containing 5 g/ml and determine the extinction at 218 and 276 nm.

$$\sum {}^{1\%}_{1\ cm\ (218\ nm)}\ 935-960;$$

$$\sum {}^{1\%}_{1\ cm\ (276\ nm)}\ 373.$$

A-1.1.2 *Calculation*

$$\text{Propyl gallate, percent} = \frac{\sum {}^{1\%}_{1cm}\ \text{of sample}}{\sum {}^{1\%}_{1cm}\ \text{of a pure standard reference}} \times 100$$

A-1.2 Bismuth Nitrate Precipitation

A-1.2.1 *Reagents*

A-1.2.1.1 *Acetone*

A-1.2.1.2 *Bismuth nitrate*

Dissolve 5 g of bismuth nitrate in 25 ml of distilled water and 25 ml of glacial acetic acid. Dilute the solution to 250 ml.

A-1.2.1.3 *Acetic acid* — 0.1 N.

A-1.2.1.4 *Nitric acid* — 0.05 N.

A-1.2.2 *Procedure*

Dry a suitable quantity of sample in an oven at 90°C for 6 hours. Cool it in a desiccator. Weigh 100 mg of octyl gallate into a 250 ml beaker. Add 15 ml of acetone and 10 ml of water and bring to the boiling point. Add 10 ml of bismuth nitrate and bring again to the boiling point. Cool to room temperature. Filter through a tared sintered glass crucible. Wash twice with acetic acid and twice with water. Wash six times with nitric acid at 80°C pressing the precipitate down well between each wash. Wash twice with water. Dry at 110°C to constant mass.

A-1.2.3 *Calculation*

$$\text{Octyl gallate, percent by mass} = \frac{\text{Mass of precipitate} \times 0.557\ 4}{\text{Mass of the sample}} \times 100$$

A-2 MOISTURE

Oven-drying and Karl Fischer methods have been specified. In case of dispute, Karl Fischer method shall be used.

A-2.1 Oven Drying

Weigh about 2 g of well-ground material in the tared dish. Place the dish containing the material in a ventilated oven at 90 ± 1°C for 6 hours. Cool the dish in a desiccator and weigh. Calculate loss on drying percent by mass.

A-2.2 Karl Fischer method as described in IS 2362 shall be used.

A-3 SULPHATED ASH

A-3.1 Reagent

A-3.1.1 Concentrated Sulphuric Acid

A-3.2 Procedure

Weigh about 2 g of the material in a tared crucible. Ignite gently until the material is thoroughly charred, cool, moisten the residue with 1 ml of sulphuric acid and ignite gently again till the carbon is completely consumed. Cool the crucible in a desiccator and weigh.

NOTE — Carry out the ignition in a place protected from air currents and use as low a temperature as possible to effect the combustion of carbon.

A-3.3 Calculation

$$\text{Sulphated ash, percent by mass} = \frac{M_1}{M_2} \times 100$$

where,

M_1 = mass in g of the residue, and

M_2 = mass in g of the material for the test.

A-4 CHLORINATED ORGANIC COMPOUNDS (AS CHLORINE)

A-4.1 Reagents

A-4.1.1 *Sodium hydroxide* — 0.1 N.

A-4.1.2 *Nitric Acid*

A-4.1.3 *Calcium Carbonate*

A-4.1.4 *Silver Nitrate* — 0.1 N.

A-4.1.5 *Hydrochrolic Acid* — 0.01 N.

B-4.2 Procedure

Dissolve 1 g of the sample in 10 ml of 0.1 N sodium hydroxide. Acidify with nitric acid solution and filter off the precipitate. Mix the precipitate with 2 g of calcium carbonate, dry the mixture and then ignite. Take up the ignition residue in 20 ml of dilute nitric acid and filter. Mix the solution with 0.5 ml of 0.1 N silver nitrate. The turbidity should not be more than that obtained in 20 ml of dilute nitric acid by addition of 0.5 ml of 0.1 N silver nitrate and 0.3 ml of 0.01 N hydrochloric acid.

A-5 FREE ACID (AS GALLIC ACID)

A-5.1 Reagents

A-5.1.1 Acetone

A-5.1.2 *Hydrochloric Acid* — 0.005 N.

A-5.1.3 *Buffer* — *p*H 5.

A-5.1.4 *Bromocresol Green*

Dissolve 50 mg of Bromocresol green in 100 ml of alcohol and fitter if necessary.

A-5.1.5 *Sodium Hydroxide* — 0.05 N.

A-5.2 Procedure

To a mixture of 50 ml of carbon dioxide-free water and 50 ml of acetone, add 5 drops of bromocresol green and titrate with 0.005 N hydrochloric acid to match a buffer containing the same amount of indicator. Dissolve 0.4 g of the sample in 50 ml of acetone and add 50 ml of carbon dioxide-free water, 5 drops of bromocresol green and the amount of 0.005 N hydrochloric acid found in the preliminary test to bring the solvent to *p*H 5. Titrate the solution back to *p*H 5 with 0.05 N sodium hydroxide, matching against the buffer. Each ml of 0.05 N sodium hydroxide is equivalent to 8.506 mg of gallic acid.

A-6 SPECIFIC ABSORPTION

Prepare one percent solution of the sample in ethanol and find out its specific absorption in a suitable spectrophotometer using 1 cm cell at 275 nm.

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 1986 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards Monthly Additions'.

This Indian Standard has been developed from Doc: No. FAD 8 (720).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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